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ULTRAHIGH-VACUUM CREEP BEHAVIOR  
OF COLUMBIUM AND TANTALUM ALLOYS  
AT 2000° AND 2200° F FOR  
TIMES GREATER THAN 1000 HOURS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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# ULTRAHIGH-VACUUM CREEP BEHAVIOR OF COLUMBIUM AND TANTALUM ALLOYS AT 2000<sup>0</sup> AND 2200<sup>0</sup> F FOR TIMES GREATER THAN 1000 HOURS

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## SUMMARY

Creep tests of 1000 hours and greater were conducted at 2000<sup>0</sup> and <sup>All</sup>2200<sup>0</sup> F on six commercially available refractory alloys at pressures of 10<sup>-8</sup> to 10<sup>-9</sup> torr. The creep behavior of three columbium alloys, <sup>Cb</sup>FS-85 (Cb + 28Ta + 10W + 1Zr), <sup>Cb</sup>D-43 (Cb + 10W + 1Zr + 0.1C), and <sup>Cb</sup>Cb-752 (Cb + 10W + 2.5Zr), and three tantalum alloys, <sup>Ta</sup>T-222 (Ta + 9.6W + 2.4Hf + 0.01C), <sup>Ta</sup>T-111 (Ta + 8W + 2Hf), and <sup>Ta</sup>Ta-10W (90Ta + 10W) was determined at stress-to-density ratios of 2.6×10<sup>4</sup> and 1.3×10<sup>4</sup> inches, respectively, at 2000<sup>0</sup> and 2200<sup>0</sup> F. Comparison of creep behavior on the density-compensated basis shows that the tantalum alloy T-222 was the most creep resistant material evaluated. A total creep strain of only 0.1 percent resulted from testing this alloy for 5000 hours at 2000<sup>0</sup> F at a stress of 15 900 pounds per square inch; while at 2200<sup>0</sup> F and 8000 pounds per square inch, 0.65-percent strain occurred in 5000 hours. The relative order of creep resistance, on a density-compensated basis, was the same for the materials evaluated at 2000<sup>0</sup> and 2200<sup>0</sup> F: T-222, Ta-10W, D-43, FS-85, T-111, and Cb-752.

## INTRODUCTION

In advanced Rankine cycle, space-electric-power systems of the nuclear turbogenerator type, highly fabricable refractory materials such as tantalum and columbium alloys are of interest as containment materials for liquid alkali metals (refs. 1 and 2). In such applications these alloys, under stress, will be subjected to very longtime exposures (10 000 hr or longer) to liquid metals at relatively high temperatures (1800<sup>0</sup> to 2400<sup>0</sup> F). Under these conditions, creep resistance of the containment material becomes an important design consideration. Unfortunately, very little pertinent information on the longtime creep properties of candidate materials is currently available. The available creep data for columbium alloys are reviewed in references 3 and 4, which summarize longtime

creep test programs being conducted by NASA Lewis Research Center, Thompson Ramo Wooldridge (refs. 5 to 8), and Oak Ridge National Laboratory (refs. 9 and 10).

At the NASA Lewis Research Center, a limited investigation of the high-temperature creep behavior of nine columbium alloys was previously conducted (ref. 1). The study included a comparatively shorttime (approx 300 hr) creep screening program conducted at a pressure of  $10^{-7}$  torr to identify quickly the more promising alloys. Of the columbium alloys evaluated, four (FS-85, D-43, B-66, and Cb-752) were selected for further creep testing and screening in ultrahigh-vacuum creep units designed and built to achieve very low pressures ( $10^{-9}$  torr range). Subsequently, three tantalum alloys (Ta-10W, T-111, and T-222) were also selected for evaluation of their longtime, ultrahigh-vacuum creep behavior.

The high temperature creep behavior of these several columbium and tantalum alloys has been evaluated in the ultrahigh-vacuum creep units for durations in excess of 1000 hours at  $2000^{\circ}$  and  $2200^{\circ}$  F.

## MATERIALS

The test materials evaluated in this study were procured in the form of sheets 0.030 inch thick and generally 12 inches wide by 24 inches long. The results of chemical analyses of the as-received sheet material are listed in table I.

The processing schedules used to produce the sheet were selected by the producers, and varied for the materials evaluated. In general, the effects of processing variables on creep properties were not evaluated in the present study; however, for one alloy, D-43, material from two sheets was evaluated, the material being from different ingots and the sheets having been produced by different working and annealing schedules. The so-called "optimum processing" procedure used for one of the sheets of D-43 evaluated herein is described in reference 11. Available information on the processing history of the evaluated materials is given in the appendix.

Creep specimens, having a 1-inch gage length and a 1/4-inch gage width as shown in figure 1, were machined from the as-received sheet with the specimen axis parallel to the final rolling direction. These were generally evaluated in the recrystallized condition to minimize effects due to differing amounts of cold work. The recrystallization treatment employed was a 1-hour vacuum ( $10^{-6}$  torr) anneal approximately  $200^{\circ}$  F above the temperature required to produce complete recrystallization in 1 hour. The recrystallization temperature used and the resulting hardness and grain size are presented in table II. This heat treatment resulted in a fine equiaxed grain structure for all alloys except D-43 (heat X110-322). In this latter alloy, recrystallization had taken place but with greatly

increased growth in the direction parallel to the final rolling direction. Figure 2 shows the microstructure resulting from the recrystallization anneals used. It is realized that for the majority of these materials a fine-grained structure may not possess the maximum creep strength; however, a detailed investigation of the effect of microstructure on creep properties was beyond the scope of this experiment.

All creep specimens were weighed to the nearest 0.1 milligram before and after the recrystallization anneal in an attempt to note any possible contamination resulting from heat treating prior to creep testing. In all cases no significant weight changes were observed; that is, the total contamination observed would amount to a maximum of only 15 parts per million by weight.

## APPARATUS

The creep facilities used in this study have been described previously in references 1 and 2. The major features of one such ultrahigh vacuum creep unit are shown in figure 3. Briefly, the unit consists of a bakable stainless-steel chamber, which is evacuated by a 400-liter-per-second sputter ion pump (to pressures of  $10^{-8}$  torr and lower) and utilizes tungsten rods for internal deadweight loading. Power is supplied to a tubular tantalum heater by a 15-kilowatt saturable-core reactor. Temperature is measured by a platinum-13-percent rhodium/platinum thermocouple and is controlled by a proportioning type controller. Pressure is measured by means of a nude hot-cathode ionization gage.

## PROCEDURE

The procedure for preparing the sheet specimens is identical to that previously described in reference 2. Fiducial marks in the form of Knoop hardness impressions are placed 1.000 inch apart in the center of the reduced section of the creep specimen. Generally, three platinum-13-percent rhodium/platinum thermocouples are tied to the reduced section of the creep specimen, one at the gage center and the remaining ones at the extremes of the gage section. The temperature gradient along the gage section was less than  $6^{\circ}$  F. The entire reduced section of the creep specimen is wrapped with 2-mil tantalum foil to reduce contamination from outgassing of the chamber and to shield the thermocouples from direct radiation from the heater. Sight holes were cut in the foil such that the fiducial marks were visible. After insertion of the specimen into the creep chambers, the direct-weight load is supported on a pedestal during leak checking with a helium mass spectrometer, bakeout of the chamber and pump, and heating of the speci-

men to test temperature.

Two days are required to bring the specimen from ambient to test temperature. During the first day the temperature is raised slowly to approximately 1200° F while maintaining a pressure below the mid 10<sup>-7</sup> torr range. The assembly is allowed to outgas overnight; then the specimen is brought to the desired test temperature and allowed to soak 1 hour prior to applying the load. Generally, the pressure is in the low 10<sup>-8</sup> torr range within 20 hours from the start of test and continues to decrease slowly as testing proceeds. The variation of pressure for a typical 1000-hour test at 2000° F is shown in figure 4. As a means of checking temperature stability as indicated by the thermocouples, surface brightness temperature is measured at the fiducial marks prior to loading and at the end of testing with a micro-optical pyrometer.

## Creep-Strain Measurement

Specimen strain was measured optically by means of a cathetometer clamped to the vacuum chamber frame. The precision of creep-strain measurements is estimated to be ±0.04 percent for the 1-inch gage length used. In all instances an initial gage length was read at the test temperature prior to loading the specimen. The strain on loading was measured and is incorporated in the reported total creep strain. Generally, this initial strain was less than 0.05 percent under the low stresses used in this study.

## Selection of Test Conditions

All creep tests reported herein were conducted at 2000° or 2200° F. Stress levels were initially selected based on the results of a preliminary creep-screening program (ref. 1) and for one alloy, FS-85, using the Manson-Haferd time-temperature parameter previously determined for this material (ref. 2). From the available information, the alloy FS-85 was initially evaluated at 2000° F and at a stress of 10 000 pounds per square inch, this combination being selected to yield an estimated creep strain of 1 percent in 1000 hours. For purposes of comparison, all other columbium alloys were also evaluated at 2000° F and at a stress of 10 000 pounds per square inch. In addition, selected alloys (both columbium- and tantalum-base alloys) were evaluated at stresses such that comparison could be made at a common stress-to-density ratio.

## Post-Test Examination

After creep testing, the specimens were reweighed and hardness readings taken on

the major surface. Sections were cut from the gage length for both metallographic and chemical analyses. The latter data were determined primarily to indicate the extent of any changes in oxygen content during testing as a result of reaction with the test environment.

## RESULTS AND DISCUSSION

### Columbium Alloys

Creep curves for the alloys FS-85, D-43, B-66, and Cb-752 at 2000° F under a stress of 10 000 psi are shown in figure 5. Data for Cb-1Zr, a low strength but highly fabricable columbium-base alloy that has received consideration for use as tubing in advanced space-power systems, is included for purposes of comparison. The times for these alloys to reach total creep strains of 1, 2, and 5 percent under these test conditions are shown in table III.

It is apparent from figure 5 and table III that, under the indicated test conditions, alloys FS-85 and D-43 (heat 43-387, the "optimum-processed" material) have much better creep resistance than do the other alloys. It is also of interest to note the large differences in the creep behavior of the D-43 alloy in the several conditions evaluated. The "optimum-processed" material from heat 43-387, solution annealed at 3000° F during processing, was significantly more creep resistant than material from heat X110-322, which was stress relieved at 2200° F during processing. Creep data for this latter material (that tested in the 2200° F stress-relieved condition) are in excellent agreement with the results of Stephenson (ref. 9) on similar material produced prior to development of the optimum processing schedule for D-43. Although minor differences in chemistry (see table I) may be partially responsible for the differences in creep behavior of the two sheets of D-43 evaluated in this study, <sup>however</sup> it is believed that the primary factor responsible for the superior creep resistance of heat 43-387 is the more favorable carbide distribution and perhaps grain structure effected by the 3000° F inprocess anneal. In this regard, it should be noted that the manufacturer's "optimum-processing" schedule was not devised specifically to optimize longtime creep strength. Other processing schedules might well produce material with better longtime creep resistance.

On the basis of the creep behavior indicated in figure 5 and from considerations of fabricability, weldability and liquid metal corrosion resistance, it was decided to limit further creep evaluation of the columbium alloys to only three materials, FS-85, D-43 (heat 43-387) and Cb-752. Although the latter alloy was not considered promising from the standpoint of creep resistance, its higher zirconium content (nominal 2.5 percent as compared to about 1 percent for FS-85 and D-43) suggested it may be attractive from the

standpoint of liquid alkali metal corrosion resistance and was the basis for its retention in the creep program.

Although the alloy FS-85 had the best creep resistance at 2000° F when all alloys were compared at a stress of 10 000 pounds per square inch, it has considerably higher density than D-43 or Cb-752 because of its high tantalum content (see table I). Since weight is usually an important consideration in aerospace systems, it was of interest to compare the alloys at a common stress-to-density ratio rather than at constant loads only. For this purpose, additional creep tests of D-43 (heat 43-387) and Cb-752 were run at 2000° F at a stress level of 8520 pounds per square inch, corresponding to a stress-to-density ratio of  $2.61 \times 10^4$  inches, which is equivalent to 10 000 pounds per square inch for FS-85. The resultant creep curves are shown in figure 6(a). Similar tests were also conducted at 2200° F, all three alloys being evaluated at a stress-to-density ratio of  $1.31 \times 10^4$  inches. The resultant creep curves are shown in figure 6(b). Minimum creep rates calculated from the linear portions of the creep curves are tabulated in table IV.

It is apparent that when these three alloys are compared at the indicated stress-to-density ratios, FS-85 and D-43 exhibit similar creep properties; both, of course, are significantly more creep resistant than Cb-752. At 2000° F, the creep curves for FS-85 and D-43 are virtually identical for the first 500 hours; at longer times, D-43 has an advantage inasmuch as its creep rate remains linear while that of FS-85 begins to accelerate slowly as shown in figure 6(a). At 2200° F the same trend is observed (fig. 6(b)). Both alloys have similar creep properties for the first 1000 hours of test, but at longer times D-43 exhibits a lower linear creep rate than FS-85.

### Tantalum Alloys

Creep curves for the tantalum alloys Ta-10W, T-111, and T-222 at 2000° F and a stress level of 15 900 pounds per square inch are shown in figure 7(a). Similar curves for tests conducted at 2200° F at a stress level near 7900 pounds per square inch are shown in figure 7(b). The stress levels were selected to permit direct comparison with the columbium alloys at common stress-to-density ratios.

The curves in figures 7(a) and (b) show that the alloy T-222 possesses outstanding creep resistance at 2000° and 2200° F and is greatly superior to both Ta-10W and T-111. For example, at 2000° F and a stress of 15 900 pounds per square inch, the T-222 alloy showed only 0.10 percent creep strain after a test duration of 5000 hours. In comparison, Ta-10W and T-111 exhibited 1.0 percent creep strain at 1250 and 350 hours, respectively. At both 2000° and 2200° F, the Ta-10W alloy was more creep resistant than T-111. Minimum creep rates for the tantalum alloys calculated from the linear portions of the creep curves are also tabulated in table V.



In view of the large differences in creep strength of the three tantalum alloys and their similarity in composition, it is of interest to attempt to correlate creep strength with composition. It appears probable that the superior creep resistance of the T-222 alloy is associated with the interaction of the reactive metal addition (hafnium) with the comparatively high interstitial content of this alloy to form a stable dispersion of hafnium carbide and/or oxide. Although the hafnium content of the T-111 evaluated in this study was higher than that of the T-222 sheet, the former alloy has such a very low interstitial content that little dispersion strengthening is possible. At low interstitial levels, the substitution of hafnium for part of the tungsten present in the Ta-10W alloy results in a decrease in creep strength, as indicated by the fact that the T-111 alloy is weaker than Ta-10W. This may reflect the ability of tungsten to increase the melting point of tantalum and thus lower self-diffusion rates, while hafnium in solid solution in tantalum lowers the melting point and may consequently increase self-diffusion rates.

### Comparison of Creep Properties of Columbium and Tantalum Alloys on Density-Compensated Basis

In order to facilitate a comparison of the creep properties of the columbium alloys FS-85, D-43, and Cb-752 with those of the tantalum alloys T-222, T-111, and Ta-10W, creep curves for tests at 2000° and 2200° F and at stress-to-density ratios of  $2.6 \times 10^4$  and  $1.3 \times 10^4$  inches, respectively, are shown in figures 8(a) and (b). Times to achieve 1-percent total creep strain under these conditions are indicated in table VI.

It is apparent that the tantalum alloy T-222 is by far the most creep resistant of the materials evaluated in this study, both at 2000° and 2200° F. Comparison of columbium- and tantalum-base alloys at a common stress-to-density ratio severely penalizes the much more dense tantalum-base alloys and indicates somewhat surprisingly that a tantalum-base alloy exhibits superior creep resistance at a temperature as low as 2000° F. From the creep data shown, the alloy Ta-10W also appears to be competitive with Cb alloys at 2000° and 2200° F. A concurrent study, however, of the alkali metal corrosion resistance of these alloys (ref. 12) indicates that the Ta-10W alloy does not have adequate corrosion resistance to liquid potassium at high temperatures, probably because of the absence of a reactive metal such as hafnium or zirconium in its composition.

### Extent of Contamination During Creep Testing

In spite of the ultrahigh-vacuum ( $10^{-8}$  to  $10^{-9}$  torr) test environments utilized for

the creep tests reported herein, previous results (ref. 2) indicated that some minor contamination of the specimens with oxygen was to be expected. The extent of such contamination in the present tests is shown in table VII. With the exception of the one specimen of D-43, which picked up 196 parts per million of oxygen during the test, the changes of oxygen levels indicated are quite small and are believed unlikely to have had any significant effect on the observed creep behavior. The results of vacuum fusion analyses indicated a decrease in oxygen content for the Cb-752 alloy; the mechanism of this loss is not yet understood.

Metallography

and photo tables III, IV, + V

Portions of the reduced gage section of creep-tested specimens were examined metallographically. Photomicrographs of typical changes in microstructure that were observed are presented for two columbium alloys, FS-85 and D-43, and for one tantalum alloy, T-111.

FS-85. - Comparison of photomicrographs of the FS-85 specimen creep tested at 2200° F and 5000 pounds per square inch for 3192 hours with the untested material (figs. 9(a) and (b), respectively) shows an increase in the amount of second phase present with no noticeable change in grain size. The formation of the large, unidentified second-phase particle was accompanied by softening. Vickers hardness number (10 kg load) decreased from 189 for the pretest condition to 177 after the 2200° F creep test.

D-43 (heat 43-387). - Comparison of photomicrographs of the D-43 (heat 43-387) specimen creep tested at 2200° F and 4270 pounds per square inch for 1848 hours with the untested material (figs. 10(a) and (b), respectively) shows that grain growth accompanied with changes in carbide morphology has taken place.

The elongated grain structure initially present was eliminated by the formation of equiaxed grains. The morphology of the dispersed carbide phase changed considerably. Prior to testing, an acicular precipitate oriented parallel to the final rolling direction existed; whereas after creep testing, the precipitate appears spherical in shape and randomly dispersed.

This grain growth and spheridization of particles was accompanied by a decrease in hardness. Vickers hardness number (10 kg load) decreased from 198 (pretest condition) to 165.

T-111. - Comparison of photomicrographs of the T-111 specimen creep tested at 2000° F and 15 900 pounds per square inch for 1848 hours with the untested material (figs. 11(a) and (b), respectively) shows the formation of large, unidentified second-phase particles with no change in grain size. The particles appear to be aligned normal to the stress axis. Examination at higher magnification revealed that the particles were pre-

dominately in the grain boundaries and that no void formation had taken place. Vickers hardness number (10 kg load) showed essentially no change after creep testing (206 initially, 209 after creep testing).

## SUMMARY OF RESULTS

The longtime, high-temperature creep behavior of several commercially available, fabricable columbium- and tantalum-base alloys was evaluated under conditions of ultrahigh-vacuum ( $10^{-8}$  to  $10^{-9}$  torr). The results of this study are as follows:

1. The tantalum-base alloy, T-222, exhibited outstanding creep strength when compared with the alloys Ta-10W, D-43, FS-85, T-111, and Cb-752 on a density-compensated basis at temperatures of  $2000^{\circ}$  and  $2200^{\circ}$  F. For example, at  $2000^{\circ}$  F and 15 900 pounds per square inch ( $2.6 \times 10^4$  in.) T-222 showed only 0.1-percent total creep strain after 5000 hours, while the second most creep resistant alloy, Ta-10W, achieved 1-percent creep strain in 1250 hours. At  $2200^{\circ}$  F and a stress-to-density ratio of  $1.31 \times 10^4$  inches, T-222 exhibited 0.65-percent creep strain in 5000 hours, more creep resistant by a time factor of 8 than any other evaluated in this study.

2. Of the columbium alloys evaluated, FS-85 and D-43 were the most creep resistant. Their creep behavior was very similar in most respects. Although their minimum creep rates under the conditions evaluated were almost identical, the time period during which the rate remained linear was longer for the D-43 alloy, which suggests that it is a more structurally stable alloy than FS-85 under the imposed test conditions.

These creep results generally represent the properties of these alloys in the fine-grained, recrystallized condition. Thermal and mechanical treatments which alter the structures of these materials would be expected to change their creep resistance.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, October 6, 1965.

## APPENDIX - PROCESSING HISTORY OF EVALUATED MATERIALS

The processing history of the materials evaluated influences their metallurgical structure and thus their creep properties. Although it is not possible in this report to document completely the processing history of these alloys, some of the more important information which influences the grain size and particle morphology is given in the description that follows:

- (1) FS-85: cold rolled 50 percent following last inprocess anneal
- (2) D-43 (heat X110-322): cold rolled 60 percent following last inprocess anneal, annealed 1 hour at 2200<sup>0</sup> F prior to shipping (material not heated above 2200<sup>0</sup> F during processing)  
D-43 (heat 43-387): penultima gage sheet solution annealed at 3000<sup>0</sup> F, cold worked approximately 25 percent, then heat treated 1 hour at 2600<sup>0</sup> F (ref. 11)
- (3) Cb-752: warm-cold rolled approximately 85 percent following last inprocess anneal, annealed 1 hour at 2200<sup>0</sup> F, and flattened by stretcher leveling
- (4) B-66: warm-cold rolled approximately 70 percent following last inprocess anneal (1 hr at 2000<sup>0</sup> F), annealed 1 hour at 2000<sup>0</sup> F
- (5) Ta-10W: cold rolled approximately 90 percent following last inprocess anneal
- (6) T-111: cold rolled 80 percent following last inprocess anneal
- (7) T-222: cold rolled approximately 85 percent following last inprocess anneal, annealed 2 hours at 2400<sup>0</sup> F prior to shipping

## REFERENCES

1. Hall, Robert W.; and Titran, Robert H.: Creep Properties of Columbium Alloys in Very High Vacuum. Paper presented at Symposium on Applications of Refractory Metals, AIMME (Los Angeles), Dec. 1963.
2. Titran, R. H.; and Hall, R. W.: High-Temperature Creep Behavior of a Columbium Alloy, FS-85. NASA TN D-2885, 1965.
3. Bartlett, E. S.; and Van Echo, J. A.: Creep of Columbium Alloys. Rept. No. DMIC Memo. 170, Battelle Memorial Inst., June 24, 1963.
4. English, Jerome J.; and Bartlett, Edwin S.: Recent Information on Long-Time Creep Data for Columbium Alloys. Rept. No. DMIC Memo. 203, Battelle Memorial Inst., Apr. 26, 1965.
5. Sawyer, J. C.; and Philleo, C. H.: Generation of Long Time Creep Data of Refractory Alloys at Elevated Temperatures. Fourth Quarterly Rept., Electromechanical Div., Thompson Ramo Wooldridge, Inc. (NASA CR-54123), July 1964.
6. Sawyer, J. C.; and Philleo, C. H.: Generation of Long Time Creep Data of Refractory Alloys at Elevated Temperatures. Fifth Quarterly Rept., Electromechanical Div., Thompson Ramo Wooldridge, Inc. (NASA CR-54228), Nov. 1964.
7. Sawyer, J. C.; and Philleo, C. H.: Generation of Long Time Creep Data of Refractory Alloys at Elevated Temperatures. Sixth Quarterly Rept., Equipment Labs., Thompson Ramo Wooldridge, Inc. (NASA CR-54287), Jan. 1965.
8. Sawyer, J. C.; and Philleo, C. H.: Generation of Long Time Creep Data of Refractory Alloys at Elevated Temperatures. Seventh Quarterly Rept., Equipment Labs., TRW, Inc. (NASA CR-54393), Apr. 1965.
9. Stephenson, R. L.: Comparative Creep-Rupture Properties of D-43 and B-66 Alloys. Rept. No. ORNL-TM-944, Oak Ridge Nat. Lab., Nov. 1964.
10. McCoy, H. E.: Creep Properties of Nb-1% Zr Alloy. J. Less Common Metals, vol. 8, no. 1, Jan. 1965, pp. 20-35.
11. Clark, James S.; Mincher, A. L.; and Villee, George N.: The Development of Optimum Manufacturing Methods for Columbium Alloy Sheet. Rept. No. RTD-TDR-63-4236, E. I. du Pont de Nemours & Co., Dec. 20, 1963.
12. Scheuermann, C. M.: Corrosion of Refractory Alloys by Refluxing Potassium. Fourth AIME Symposium on Refractory Metals, French Lick, Indiana, October 3-5, 1965. (Also NASA TM X-52136, 1965.)

TABLE I. - CHEMICAL ANALYSIS AND DENSITY OF COLUMBIUM AND  
TANTALUM SHEETS PRIOR TO CREEP TESTING

Alloy	Composition, weight percent										Density, lb/in. <sup>3</sup>
	Tung- sten	Zirco- nium	Hafnium	Other	Oxygen	Carbon	Nitrogen	Hydrogen	Colum- bium	Tanta- lum	
Columbium:											
Cb-12Zr	-----	0.95	----	-----	0.0073	0.0100	0.0070	0.0003	Bal.	---	0.310
B-66	-----	1.16	----	Vanadium, 5.02 Molybdenum, 4.85	.0159	.0077	.0102	.0003	↓	---	.305
Cb-752	9.77	2.73	----	-----	.0178	.0038	.0100	.0003	↓	---	.326
FS-85	9.49	.80	----	Tantalum, 27.76	.0180	.0060	.0042	.0002	↓	---	.383
D-43 <sup>a</sup>	9.05	1.04	----	-----	.0096	.0860	.0031	.0001	↓	---	.326
D-43 <sup>b</sup>	9.59	.98	----	-----	.0132	.1074	.0040	.0003	↓	---	.326
Tantalum:											
Ta-10W	10.35	----	----	-----	0.0021	0.0016	0.0040	0.0001	---	Bal.	0.606
T-111	7.60	----	2.36	-----	.0013	.0013	.0020	.0001	---	↓	.604
T-222 <sup>c</sup>	9.11	----	1.91	-----	.0040	.0113	.0014	.0002	---	↓	.605

<sup>a</sup>Heat X110-322.

<sup>b</sup>Heat 43-387.

<sup>c</sup>When purchased alloy met manufacturer's specifications but does not meet present NASA specifications which require slightly higher tungsten and hafnium content in T-222 alloy.

TABLE II. - RESULTS OF RECRYSTALLIZATION  
ON COLUMBIUM AND TANTALUM ALLOYS

Alloy	Annealing temperature, °F	Vickers hardness (10 kg load)	Average grain diameter, mm
Cb-1Zr	2200	100	0.031
B-66	2600	239	.050
Cb-752	2600	178	.022
FS-85	2600	189	.019
D-43	<sup>a</sup> 2200	158	Worked structure
D-43	<sup>b</sup> 2600	158	
D-43	<sup>c</sup> 2600	198	
Ta-10W	2600	212	
T-111	2600	230	.018
T-222	2800	295	.016

<sup>a</sup>Heat X110-322. Heat treated by manufacturer (stress-relieved).

<sup>b</sup>Heat X110-322. Elongated grain structure retained after annealing.

<sup>c</sup>Heat 43-387. Heat treated by manufacturer; elongated grain structure retained after annealing.

TABLE III. - TIME TO REACH INDICATED CREEP STRAINS  
UNDER TEST CONDITIONS OF 2000° F AND 10 000 POUNDS  
PER SQUARE INCH IN 10<sup>-9</sup> TORR RANGE

Alloy	Total creep strain, percent		
	1	2	5
	Time, hr		
FS-85	775	1340	2160
D-43 (heat 43-387)	530	1050	(c)
D-43 (heat X110-322) <sup>a</sup>	305	510	920
D-43 (heat X110-322) <sup>b</sup>	80	250	530
B-66	200	310	(c)
Cb-752	75	125	220
Cb-1Zr	2	5	20

<sup>a</sup>Annealed 1 hr at 2600° F (10<sup>-6</sup> torr) prior to creep testing.

<sup>b</sup>Stress relieved 1 hr at 2200° F by manufacturer.

<sup>c</sup>Test terminated after achieving 2-percent creep strain.

TABLE IV. - MINIMUM CREEP RATES  
FOR COLUMBIUM ALLOYS

Alloy	Temperature, °F	
	2000	2200
	Stress-to-density ratio, in.	
	$2.60 \times 10^4$	$1.31 \times 10^4$
	Minimum creep rate, in./in./hr	
FS-85	$8.3 \times 10^{-6}$	$1.3 \times 10^{-5}$
D-43 (heat 43-387)	$8.3 \times 10^{-6}$	$1.2 \times 10^{-5}$
Cb-752	$1 \times 10^{-5}$	$1.2 \times 10^{-4}$

TABLE V. - MINIMUM CREEP RATES  
FOR TANTALUM ALLOYS

Alloy	Temperature, °F	
	2000	2200
	Stress-to-density ratio, in.	
	$2.6 \times 10^4$	$1.31 \times 10^4$
	Creep rate, in./in./hr	
T-222	$a_2 \times 10^{-7}$	$a_1.25 \times 10^{-6}$
Ta-10W	$6.4 \times 10^{-6}$	$9.4 \times 10^{-6}$
T-111	$3.8 \times 10^{-5}$	$1.1 \times 10^{-5}$

<sup>a</sup>Test still running, linear creep rate covers present duration of testing (5000 hr).



TABLE VI. - COMPARISON OF TIMES TO REACH  
1-PERCENT CREEP STRAIN FOR COLUMBIUM  
AND TANTALUM ALLOYS

Alloy	Temperature, °F	
	2000	2200
	Stress-to-density ratio, in.	
	$2.6 \times 10^4$	$1.3 \times 10^4$
	Time to achieve 1-percent creep strain, hr	
T-222	(a)	(c)
Ta-10W	1250	925
D-43 <sup>b</sup>	900	775
FS-85	775	960
T-111	350	325
Cb-752	125	85

<sup>a</sup>Test still in progress; 0.1 percent creep strain in 5000 hr.

<sup>b</sup>Heat 43-387.

<sup>c</sup>Test still in progress; 0.65 percent creep strain in 5000 hr.

TABLE VII. - RESULTS FROM VACUUM FUSION ANALYSIS FOR OXYGEN  
ON ULTRAHIGH-VACUUM CREEP SPECIMENS AFTER TESTING

Specimen	Time, hr	Temper- ature, °F	Stress, psi	Pressure, torr		Increase in oxygen content during testing, ppm
				Start of test	End of test	
Cb-1Zr-5	42	2000	10 000	$5 \times 10^{-7}$	$1.5 \times 10^{-8}$	(a)
B-66-2	384	2000	10 000	$4.2 \times 10^{-7}$	$1 \times 10^{-8}$	11
FS-85-14	2212	2000	10 000	$3 \times 10^{-7}$	$3 \times 10^{-9}$	60
FS-85-29	3192	2200	5 000	$7 \times 10^{-7}$	$6 \times 10^{-9}$	60
D-43-4 <sup>b</sup>	1000	2000	10 000	$3 \times 10^{-7}$	$4.5 \times 10^{-9}$	196
D-43-1 <sup>b</sup>	1004	2000	10 000	$2.3 \times 10^{-7}$	$3 \times 10^{-9}$	24
D-43-10 <sup>c</sup>	1146	2000	10 000	$1.6 \times 10^{-7}$	$2.4 \times 10^{-9}$	13
D-43-9 <sup>c</sup>	1990	2000	8 520	$2.5 \times 10^{-7}$	$2.7 \times 10^{-9}$	30
D-43-11 <sup>c</sup>	1848	2200	4 270	$2.5 \times 10^{-8}$	$3.9 \times 10^{-9}$	21
Cb-752-4	500	2000	10 000	$4 \times 10^{-8}$	$6 \times 10^{-9}$	-88
Cb-752-7	500	2200	4 270	$3.6 \times 10^{-7}$	$6 \times 10^{-9}$	-65
Cb-752-8	480	2000	8 520	$7.6 \times 10^{-8}$	$2.6 \times 10^{-9}$	-90
Ta-10W-4	1341	2000	15 900	$5 \times 10^{-7}$	$6.6 \times 10^{-9}$	24
Ta-10W-2	1007	2200	8 000	$1.9 \times 10^{-7}$	$7.4 \times 10^{-9}$	12
T-111-4	1484	2000	15 900	$3 \times 10^{-7}$	$3.5 \times 10^{-9}$	24

<sup>a</sup>Not analyzed. Rupture of specimen led to leak that contaminated specimen after creep test.

<sup>b</sup>Heat X110-322.

<sup>c</sup>Heat 43-387.

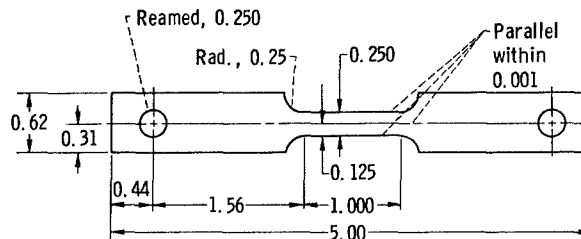


Figure 1. - Standard sheet creep-test specimen. (All dimensions are in inches.)



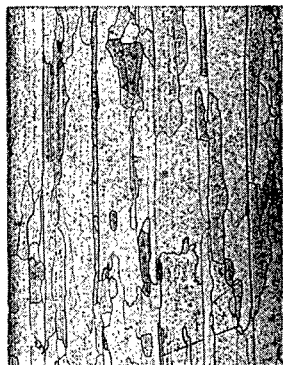
D-43 (heat X 110-322) stress relieved 1 hour at 2200° F, as received



TA-10W, annealed 1 hour at 2600° F



FS-85, annealed 1 hour at 2600° F



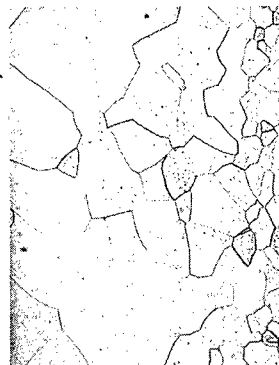
D-43 (heat X 110-322) annealed 1 hour at 2600° F at Lewis



T-111, annealed 1 hour at 2600° F



Cb-752, annealed 1 hour at 2600° F



B-66, annealed 1 hour at 2600° F



D-43 (heat 43-387) annealed 1 hour at 2600° F, as received



T-222, annealed 1 hour at 2800° F



Cb-1Zr, annealed 1 hour at 2200° F

Figure 2. - Microstructure of materials evaluated prior to creep testing. X250. (Reduced 50 percent in printing.)

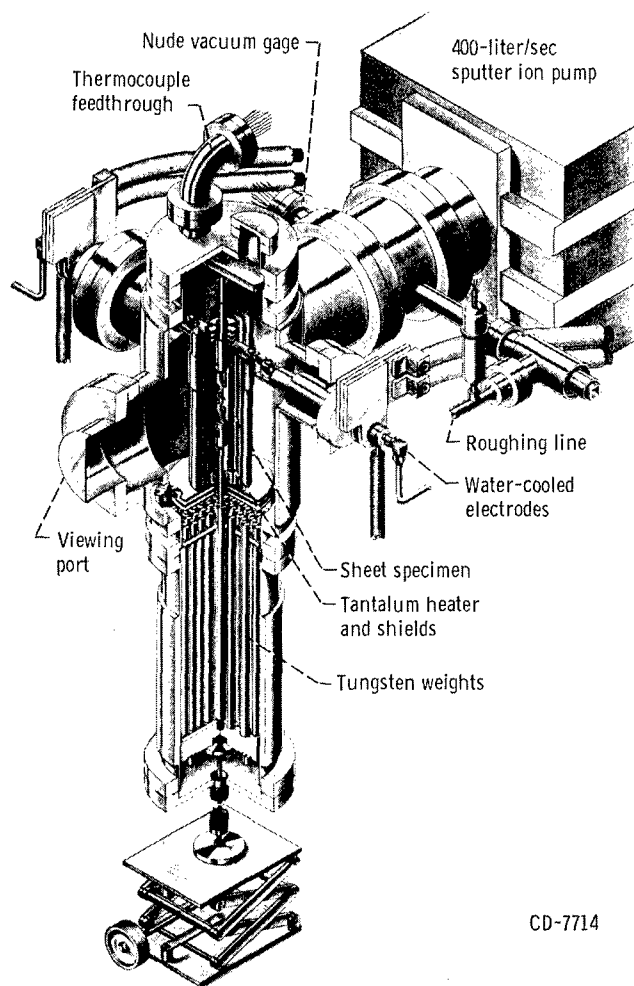


Figure 3. - Cutaway drawing of ultrahigh-vacuum creep unit.

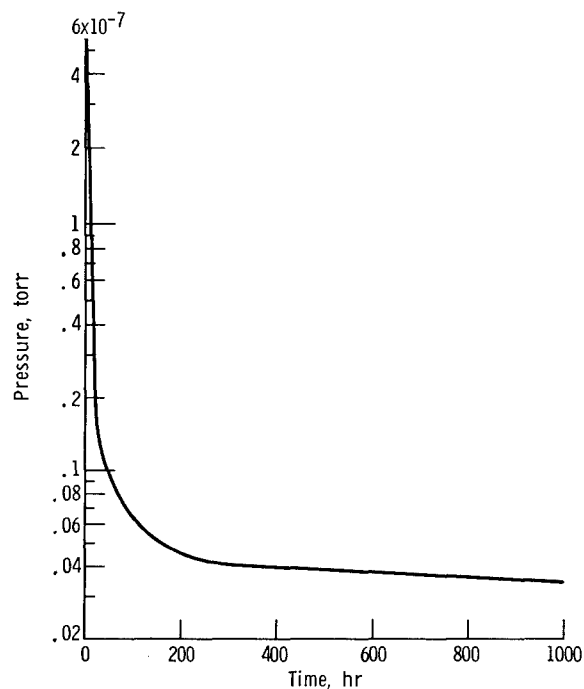


Figure 4. - Variation of pressure for typical creep test.

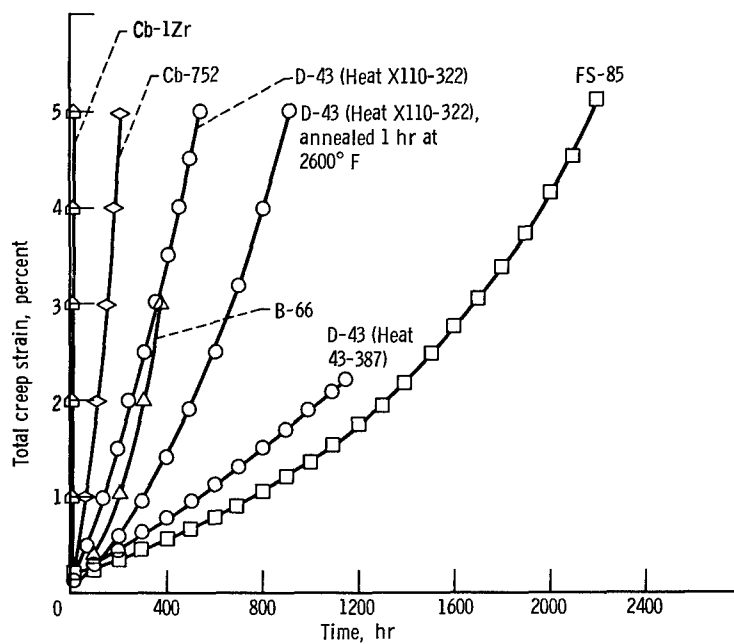
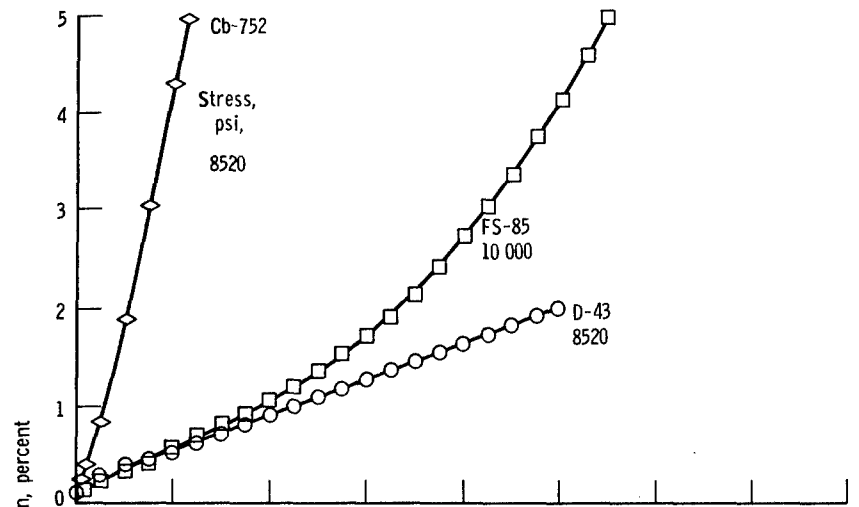
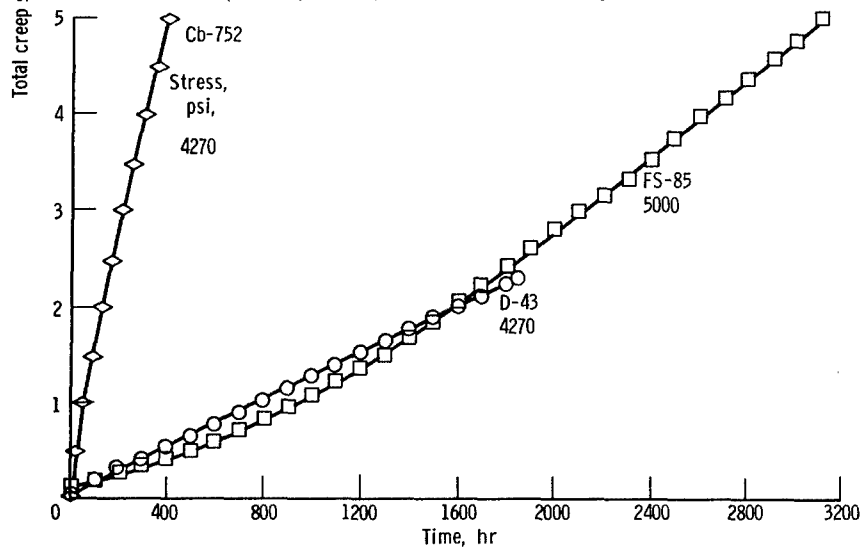


Figure 5. - Ultrahigh-vacuum creep behavior of several columbium alloys at 2000° F and 10 000 pounds per square inch.

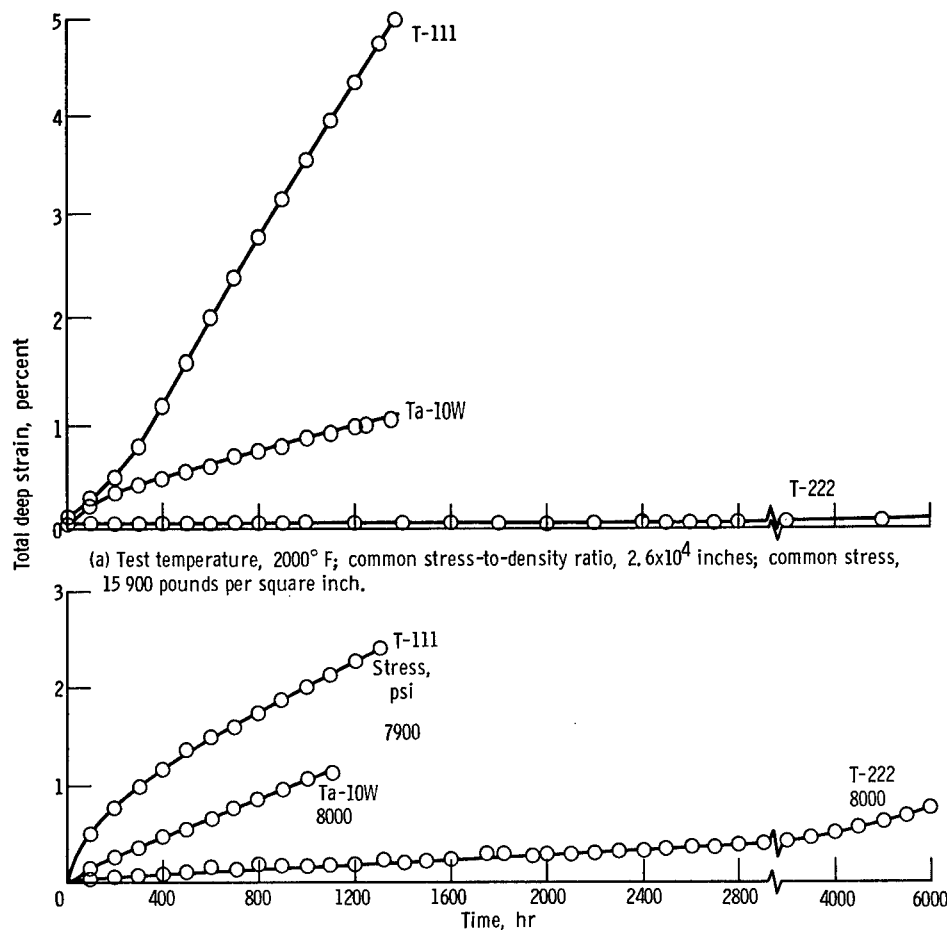


(a) Test temperature, 2000° F; common stress-to-density ratio,  $2.6 \times 10^4$  inches.



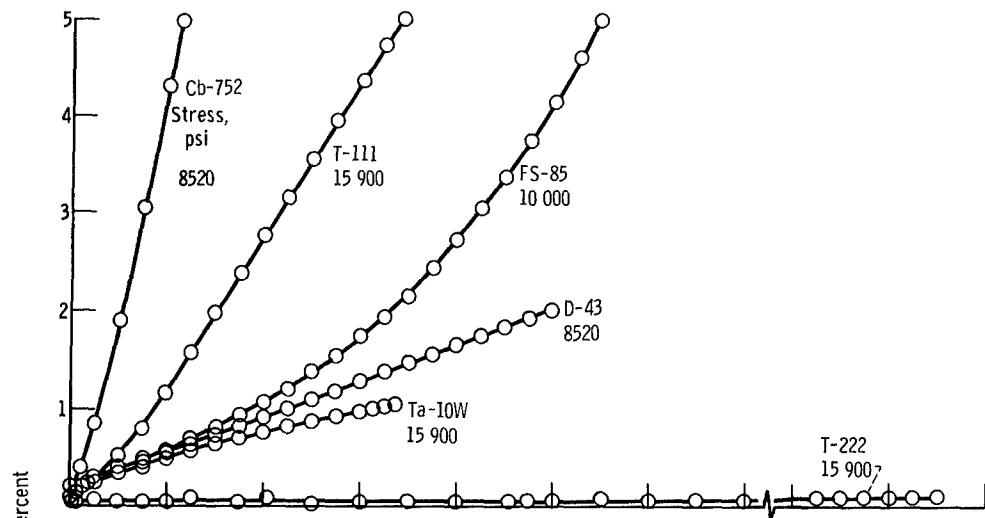
(b) Test temperature, 2200° F; common stress-to-density ratio,  $1.3 \times 10^4$  inches.

Figure 6. - Creep behavior of columbium alloys.

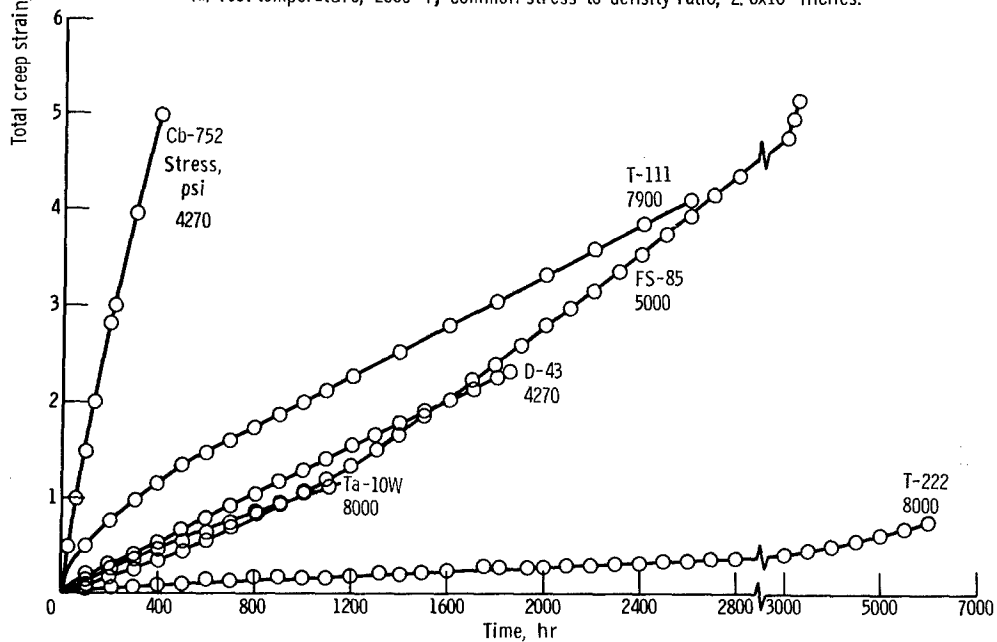


(b) Test temperature, 2200° F; common stress-to-density ratio,  $1.3 \times 10^4$  inches.

Figure 7. - Creep behavior of tantalum alloys.



(a) Test temperature, 2000° F; common stress-to-density ratio,  $2.6 \times 10^4$  inches.



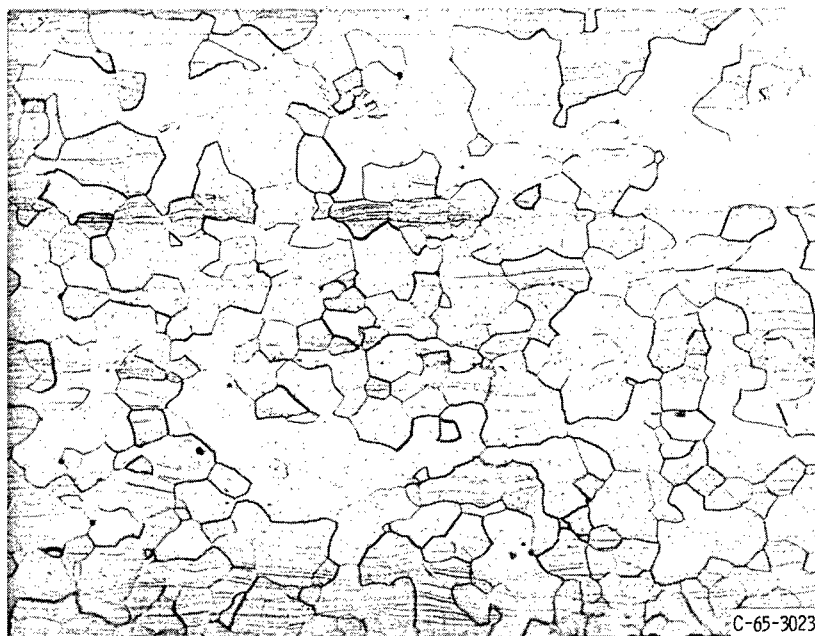
(b) Test temperature, 2200° F; common stress-to-density ratio,  $1.3 \times 10^4$  inches.

Figure 8. - Creep behavior of columbium and tantalum alloys.





(a) Creep tested at 2200° F for 3191 hours. Stress, 5000 pounds per square inch.

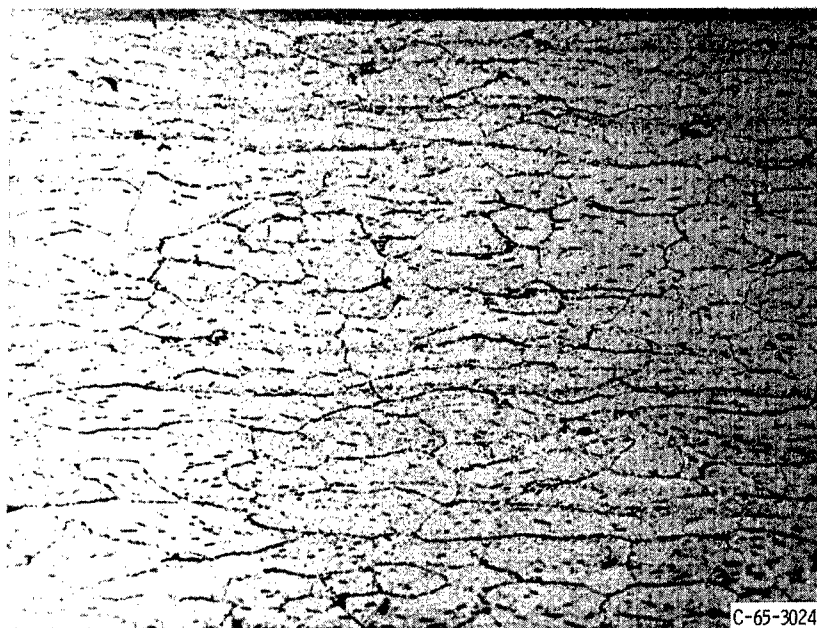


(b) Annealed 1 hour at 2600° F.

Figure 9. - Comparison of microstructure of FS-85 after creep testing with microstructure of untested material. X250.

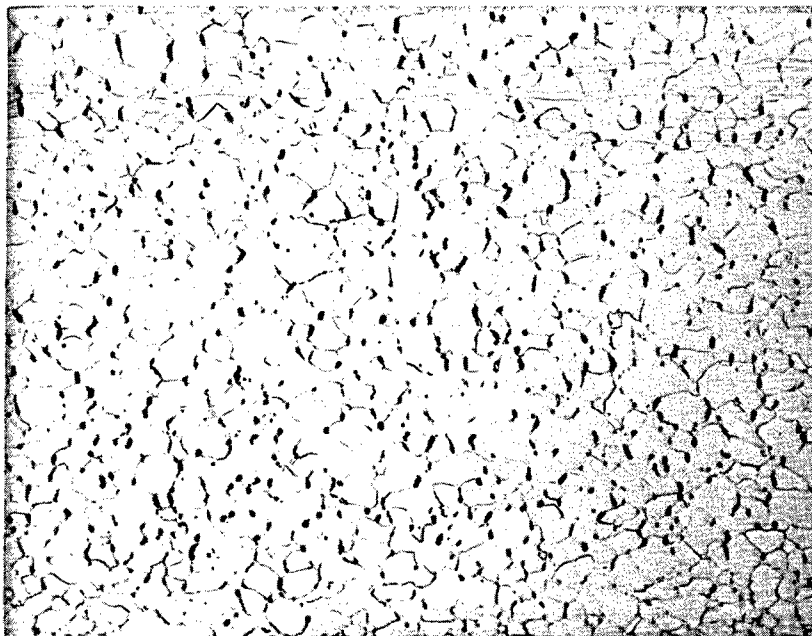


(a) Creep tested at 2200° F for 1848 hours. Stress, 4270 pounds per square inch.

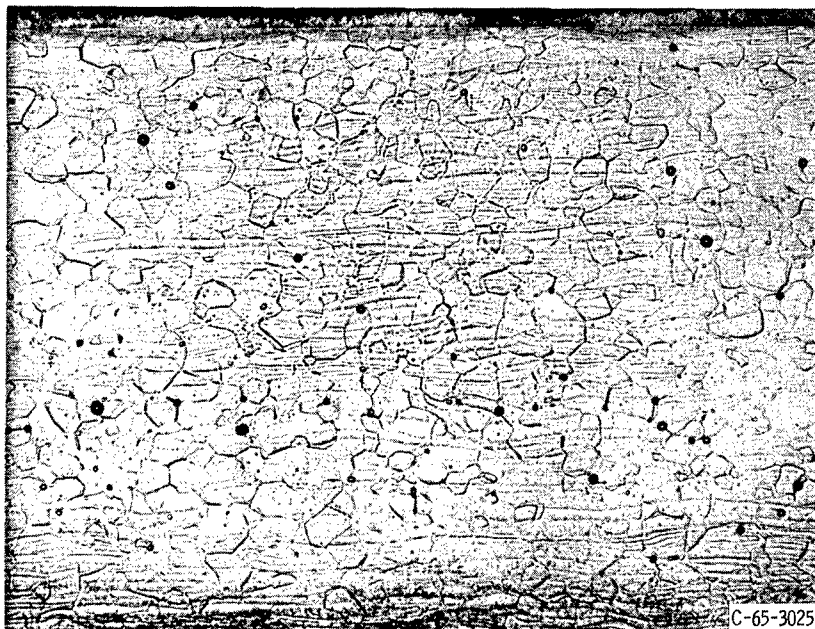


(b) Annealed 1 hour at 2600° F.

Figure 10. - Comparison of microstructure of D-43 after creep testing with microstructure of untested material. X250.



(a) Creep tested at 2000° F for 1484 hours. Stress, 15 900 pounds per square inch.



(b) Annealed 1 hour at 2600° F.

Figure 11. - Comparison of microstructure of T-111 after creep testing with microstructure of untested material. X250.